



Properties Data for Adhesion and Surface Chemistry of Aluminum

Sapphire-Aluminum, Single-Crystal Couple

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PROPERTIES DATA FOR ADHESION AND SURFACE CHEMISTRY OF ALUMINUM Sapphire-Aluminum, Single-Crystal Couple

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SUMMARY

An investigation was conducted to examine the adhesion and surface chemistry of single-crystal aluminum in contact with single-crystal sapphire (alumina). Pull-off force (adhesion) measurements were conducted under loads of 0.1 to 1 mN in a vacuum of 10^{-8} to 10^{-9} Pa ($\sim 10^{-10}$ to 10^{-11} torr) at room temperature. An Auger electron spectroscopy analyzer incorporated directly into an adhesion-measuring vacuum system was primarily used to define the chemical nature of the surfaces before and after adhesion measurements. The surfaces were cleaned by argon ion sputtering. With a clean aluminum-clean sapphire couple the mean value and standard deviation of pull-off forces required to separate the surfaces were 3015 and 298 μN , respectively. With a contaminated aluminum-clean sapphire couple these values were 231 and 241 μN . The presence of a contaminant film on the aluminum surface reduced adhesion by a factor of 13. Therefore, surfaces cleanliness, particularly aluminum cleanliness, played an important role in the adhesion of the aluminum-sapphire couples. Pressures on the order of 10^{-8} to 10^{-9} Pa ($\sim 10^{-10}$ to 10^{-11} torr) maintained a clean aluminum surface for only a short time (<1 hr) but maintained a clean sapphire surface, once it was achieved, for a much longer time.

INTRODUCTION

Clean surfaces of metal-ceramic couples adhere to one another when placed in contact in ultrahigh vacuum. A variety of methods can be employed to quantify the bonding forces. Some involve tensile pulling on the interface. The strength of adhesion is usually expressed as the force needed to pull the surfaces apart in the normal direction (i.e., the pull-off force (ref. 1)). Other methods, such as sliding friction force measurements, are based on tangential shearing of the junction (ref. 2). Both pull-off force and friction force measurements are effective in gaining quantitative information on interfacial bond strengths. The static and dynamic friction behavior of clean metal-ceramic couples is directly related to their adhesion (pull-off force) behavior in ultrahigh vacuum (refs. 3 to 5).

The first objective of the investigation was to examine the pull-off forces (adhesion forces) required to break the interfacial junctions between a single-crystal aluminum {111} pin and a single-crystal sapphire (alumina) {1011} flat surface in ultrahigh vacuum. A torsion balance adapted for in-situ pull-off force measurements from the Cavendish balance (refs. 6 and 7) was used. The second objective was to examine the surface chemistry of aluminum pin and sapphire flat surfaces by Auger electron spectroscopy (AES).

MATERIALS AND SPECIMENS

The single-crystal aluminum used as hemispherical pin specimens (fig. 1(a)) in the adhesion experiments was 99.999% pure and had $\langle 111 \rangle$ orientation. The $\{111\}$ plane was close to the interface. The radius of curvature of the pin specimen was 1.6 mm at the apex. The contacting surfaces of the aluminum pins were machined by using a diamond cutting tool and then chemically polished. Backreflection Laue photographs were taken to establish the exact orientations of the aluminum single crystals after they had been chemically polished. Specimens were less than 2° off the low-index $\langle 111 \rangle$ orientation.

The single-crystal sapphire used as flat specimens in the experiment was less than 2° off the $\langle 1011 \rangle$ orientation (fig. 1(b)). The $\{1011\}$ plane was parallel to the interface. The contacting surfaces of the sapphire flats were polished by the manufacturer and were used in the as-polished condition.

APPARATUS AND EXPERIMENT

The adhesion-measuring device used in this investigation was mounted in an ultra-high-vacuum system that contained an AES spectrometer. The mechanism for measuring adhesion was basically a pin-on-flat configuration, as shown schematically in figure 2.

The adhesion-measuring device was a torsion balance adapted from the principle of the Cavendish balance used to measure gravitational forces in 1798 (ref. 8). The adapted torsion balance consists of a solid, A, and a displacement sensor, such as an electromechanical transducer, mounted at opposite ends of a horizontal arm supported at its center by a vertical wire, such as a single-strand music wire (fig. 3). Another solid, B, is moved horizontally toward A, presses against it, and twists the wire through a small angle with a normal force—the normal loading process—thereby moving the sensor. The solid B is then gradually moved horizontally backward until the two solids are pulled apart in a normal direction—the unloading process. If the force of adhesion between the two solids is zero, A separates from B at its original position and untwists the wire, thereby moving the sensor back to its original position. If an adhesive force is present between the two solids, the force twists the wire as B moves backward until the wire develops sufficient force to separate the surfaces of A and B in the normal direction.

In this system the attractive force of adhesion and the force required to pull the surfaces of two solids apart (the pull-off force) act along a horizontal direction and are not affected by gravity and buoyancy. The axis of the weight and buoyancy of all components (arm, sensor, and wire) is different from the axis of the pull-off microforce to be measured and is in the vertical direction because of gravity. Therefore, specimen size and weight have no effect on the accuracy of measuring pull-off forces.

Since the pull-off force is measured by the torsional moment acting on the torsion wire, the force can be calibrated in three ways: (1) by calculation from the geometric shape of the torsion wire, such as its length and area of section; (2) by calculation with measured values of the natural periods of arm harmonic motion when the arm is freely oscillated; and (3) by direct comparison of the microforce to a standard weight when the arm and torsion wire are held horizontally (ref. 6). The pull-off forces determined by all three methods of calibration were nearly the same.

For the actual balance shown in figure 2 the flat specimen (corresponding to the solid A in fig. 3) was mounted on one end of a movable arm. A free-moving, rod-shaped magnetic core was mounted on the other end of the arm. The coils of a linear variable differential transformer (LVDT) were mounted on a stationary arm. There was no physical contact between the movable magnetic core and the coil structure. The movable arm was supported by a single strand of music wire acting as a torsion spring. The pin specimen (corresponding to the solid B in fig. 3) was mounted on a specimen holder attached to a manipulator.

The pin and flat specimens were rinsed with high-purity ethanol before they were placed into the vacuum chamber. After the specimens had been placed into the vacuum chamber, the system was evacuated and baked out to achieve a pressure of 7×10^{-9} Pa (5×10^{-11} torr). Then, both flat and pin specimens were argon ion sputter cleaned with a 3-keV beam at 25-mA current and an argon pressure of 0.7 mPa (5×10^{-6} torr). The ion beam was continuously rastered over the specimen surface. After sputter etching the vacuum system was reevacuated. Finally, the in-situ adhesion experiments were conducted with an ion-sputter-cleaned aluminum pin in contact with an ion-sputter-cleaned sapphire flat in ultrahigh vacuum. Also, both flat and pin specimens were analyzed by AES before and after sputter cleaning. Topographical changes can be introduced by ion bombardment for surface cleaning in the direction of increased surface roughness. Because high-purity, single-crystal aluminum and sapphire were used in this investigation, sputter-induced roughness could be minimized and its effect on adhesion was negligible. The surface roughnesses of the argon-ion-sputtered pin and flat were on the same order as those of the chemically polished aluminum pin and the as-received sapphire flat, respectively. Further, some structural damage could be imparted by the ion and electron beam to the single-crystal aluminum and sapphire surfaces. In this investigation, however, both argon-ion-sputter-cleaned aluminum pin and sapphire flat were not thermally annealed prior to adhesion experiments.

For in situ pull-off force (adhesion force) measurements in vacuum the flat specimen was brought into contact with the pin specimen by moving a motorized micrometer headscrew forward. Contact was maintained for 30 s; then the pin and flat specimen surfaces were pulled apart by moving the motorized micrometer headscrew backward. The LVDT monitored the displacement of the flat specimen. Figure 4 shows a typical force-time trace resulting from such adhesion experiments. Contact occurred at point A. The line A-B represents the region where the load was being applied. The displacement BL corresponds to the normal load. The line B-C represents the region where the contact was maintained at the given load and the specimen surfaces were stationary. The line C-D represents the region where both the unloading and separation forces were being applied on the adhesion junction. At point D the onset of separation occurred. The displacement DM corresponds to the pull-off force. After the pin specimen separated from the flat, the pin fluctuated back and forth, as represented by the E region.

RESULTS AND DISCUSSION

Surface Cleanliness

Aluminum pin.—Carbon and water are ubiquitous. Even a supposedly “clean” specimen surface will show a significant carbon and water contribution to the AES spectrum because of the presence of one or more layers of adsorbed hydrocarbons and carbon oxides. The surfaces of metals and ceramics usually contain, in addition to the constituent atoms, adsorbed films of water vapor, carbon monoxide, carbon dioxide, and oxide layers. A contaminant layer will attenuate the electron signal from the underlying surface and may mask important features in the spectrum.

Figure 5(a) presents an AES spectrum of a chemically polished, single-crystal aluminum pin surface in vacuum. A carbon contamination peak is evident as well as an oxygen peak. The carbon peak is similar to that obtained for amorphous carbon. The aluminum and oxygen peaks in figure 5(a) indicate that the surface was covered with aluminum oxide as well as a simple adsorbed oxygen film. In addition to the major AES peaks the chemically polished aluminum surface could contain small amounts of contaminant species, such as silicon, argon, nitrogen, iron, and zinc.

In a vacuum environment sputtering with rare gas ions can remove contaminants adsorbed on the surfaces of materials. Figure 5(b) presents the AES spectrum taken after the aluminum pin had been argon ion sputter cleaned. After argon ion sputtering the contamination peaks became very small, and the relative peak intensity of aluminum increased markedly. Each contaminant was less than 1%, on the order of typical AES trace capability (0.1%).

Sapphire flat.—Figures 6(a) and (b) compare AES spectra for the as-received sapphire flat specimen and the argon-ion-sputter-cleaned sapphire flat specimen. Argon ion sputtering removed the principal carbon contaminants from the sapphire flat specimen. The cleaned surface consisted of aluminum oxide and small amounts of carbon and implanted argon. The contaminants were on the order of typical AES trace capability.

Influence of Ultrahigh Vacuum on Surface Cleanliness

Clean surfaces in a vacuum environment have a certain degree of unsaturation, such as dangling bonds. They tend to accumulate gases (i.e., adsorption takes place on the surfaces). The degree of surface unsaturation can vary widely. When a high degree of unsaturation exists on a surface, certain adsorbed species will tend to chemically bond to the surface (i.e., chemisorption takes place). The surfaces of many substances are inert to certain gaseous species. When the gaseous species adsorb on these surfaces, physical or van der Waals adsorption occurs. Figures 7(a) and (b) present some of the adsorption data on an aluminum pin and a sapphire flat obtained from AES element analyses conducted using a 3-keV electron beam.

Aluminum pin.—In figure 7(a) adsorption of residual gases to an argon-ion-sputter-cleaned aluminum pin is plotted as a function of exposure time to ultrahigh vacuum (6.0×10^{-8} to 5.9×10^{-9} Pa; 4.5×10^{-10} to 4.4×10^{-11} torr) and at room temperature (23 °C). The total surface exposure after 500 hr was approximately 1.2×10^{-2} Pa-s (9.0×10^{-5} torr-s). Figure 7(a) indicates that common contaminants, such as carbon and oxygen, were easily adsorbed on a clean aluminum pin surface exposed to ultrahigh vacuum. The concentrations of carbon and oxygen increased with an increase in exposure time. Pressures on the order of 10^{-8} to 10^{-9} Pa ($\sim 10^{-10}$ to 10^{-11} torr) maintained the clean aluminum surface for only a short time (<1 hr).

Sapphire flat.—Figure 7(b) indicates that the concentrations of various constituents on the surface of an argon-ion-sputter-cleaned sapphire flat exposed to ultrahigh vacuum (1.8×10^{-8} to 8.3×10^{-9} Pa; 1.4×10^{-10} to 6.2×10^{-11} torr) all remained constant during the entire exposure time of 525 hr (i.e., surface exposure of approximately 1.7×10^{-2} Pa-s (1.3×10^{-4} torr-s)).

Adhesion

Clean aluminum in contact with clean sapphire.—Pull-off force measurements were conducted with an argon-ion-sputter-cleaned aluminum pin in contact with an argon-ion-sputter-cleaned sapphire flat in ultrahigh vacuum. Figure 8 presents the pull-off force, which reflects interfacial adhesion, as a function of exposure time after argon ion sputter cleaning for the aluminum-sapphire, single-crystal couple at pressures from 6.0×10^{-8} to 5.9×10^{-9} Pa (4.5×10^{-10} to 4.4×10^{-11} torr) and at room temperature (23 °C). Although element analysis was performed after each pull-off force measurement, repeated contacts of the same spot on the pin were made to match as closely as possible with the same contact spot on the flat. The element concentration data from AES analyses are plotted in figure 7(a) for the aluminum pin as a function of exposure time to ultrahigh vacuum after argon ion sputter cleaning.

Figure 8 indicated that when the argon-ion-sputter-cleaned aluminum pin was brought into contact with the argon-ion-sputter-cleaned sapphire flat, strong bonds formed between the two materials. Although the pull-off force was 2115 μ N at the first contact in figure 8, the mean and standard deviation of the pull-off forces at the first contacts for argon-ion-sputter-cleaned aluminum-sapphire, single-crystal couples were 2754 and 574 μ N, respectively. This high-adhesion situation applied to some degree in the repeated contacts, where fresh surfaces were continuously exposed by the repeated contact action. Although some pull-off forces, particularly data points labeled A, B, and C, were low in the repeated contacts, most remained high, as shown in figure 8. The mean value and standard deviation of the high-pull-off-force data were 3015 and 298 μ N, respectively. The low-pull-off-force data points labeled A, B, and C were not included in these calculations.

The low-pull-off-force data points labeled A and C may have been affected by surface contamination of the aluminum pin exposed to ultrahigh vacuum for long times after the previous pull-off force measurements. For example, as shown in figure 8, the pull-off force measurement at point A was performed after an interval of 74.5 hr. However, when repeated contacts were made at short intervals, the pull-off force increased from A to B and then to the high value around 3000 μ N. Under actual conditions of repeated contacts and pull-offs at short intervals, the pull-off force may not be sensitive to the recontamination of aluminum (fig. 7(a)) because the aluminum microasperities in contact were plastically deformed and their cohesive bonds fractured. Therefore, strong bonds between aluminum and sapphire are unavoidable in repeated contacts at short intervals in ultrahigh vacuum.

Contaminated aluminum in contact with clean sapphire.—Figure 9 presents the pull-off force measured for a chemically polished aluminum pin (fig. 5(a)) in contact with an argon-ion-sputter-cleaned sapphire flat (fig. 6(b)) in ultrahigh vacuum (6.3×10^{-9} Pa; 4.7×10^{-11} torr) and at room temperature (23 °C). The pull-off force is plotted as a function of the number of repeated contacts. Pull-off forces (adhesion) for the contaminated aluminum-clean sapphire couple remained low. The mean value and standard deviation of these pull-off forces were 231 and 241 μ N, respectively.

Comparing figure 9 with figure 8 shows that adhesion with the chemically polished aluminum pin was generally much lower than that with the argon-ion-sputter-cleaned aluminum pin, although pull-off force data points labeled D and E in figure 9 were similar to those labeled A and C in figure 8. The adhesion for the clean aluminum-clean sapphire couple was greater than that for the contaminated aluminum-clean sapphire couple by a factor of 13. Thus, the contaminant film on the aluminum pin surface greatly reduced adhesion.

SUMMARY OF RESULTS

The following results were obtained from this investigation of the adhesion and surface chemistry of single-crystal aluminum in contact with single-crystal sapphire:

1. For a clean aluminum-clean sapphire couple the mean value and standard deviation of pull-off forces required to separate the surfaces in contact were 3015 and 298 μ N, respectively. For a contaminated aluminum-clean sapphire couple the mean value and standard deviation of pull-off forces were 231 and unnecessarily short line 241 μ N. The presence of a contaminant film on an aluminum surface reduced adhesion by a factor of 13.
2. Pressures on the order of 10^{-8} to 10^{-9} Pa ($\sim 10^{-10}$ to 10^{-11} torr) maintained a clean aluminum surface for only a short time (<1 hr).
3. Pressures on the order of 10^{-8} Pa ($\sim 10^{-10}$ torr) or lower maintained a clean sapphire surface, once it was achieved, for a much longer time.

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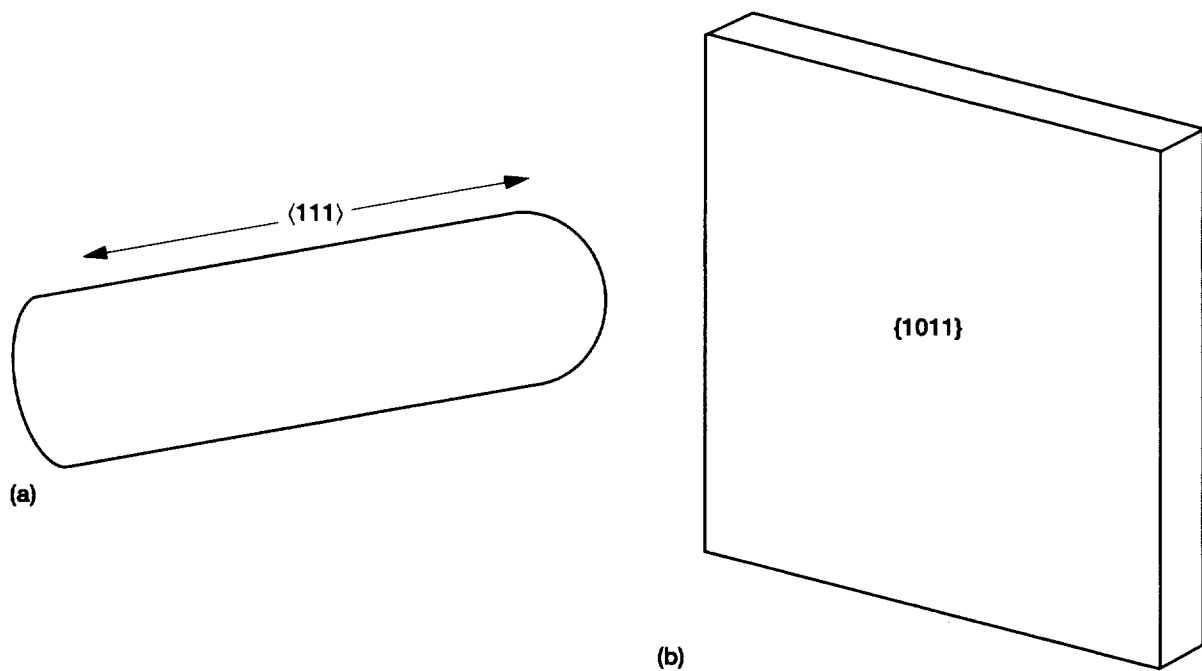


Figure 1.—Aluminum pin and sapphire flat specimens. (a) Hemispherical aluminum pin: 1.6-mm radius of curvature at apex; 12.7 mm long; and 3.2 mm in diameter. The pin axis is oriented along $\langle 111 \rangle$ direction. (b) Sapphire flat: 9 mm by 9 mm; 1.27 mm thick.

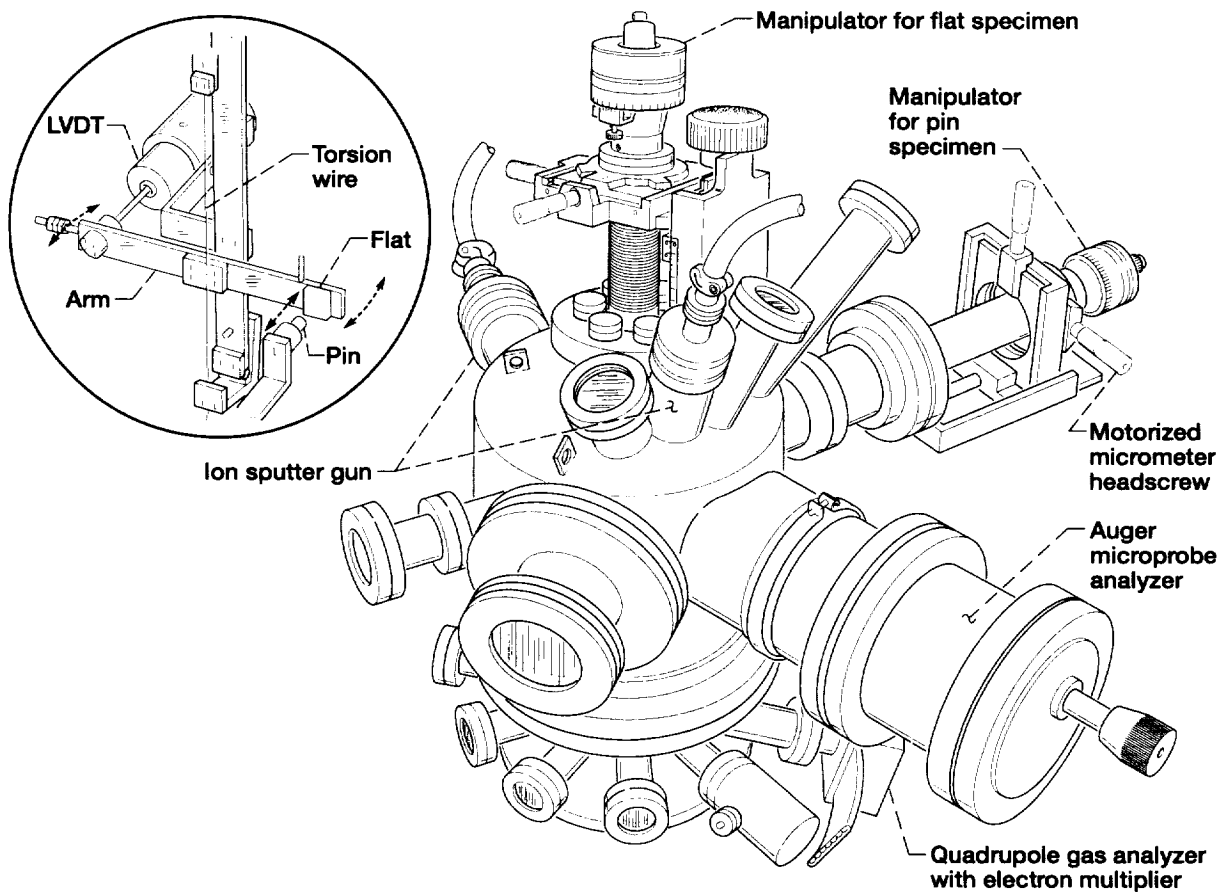


Figure 2.—Apparatus for measuring adhesion and friction in ultrahigh vacuum.

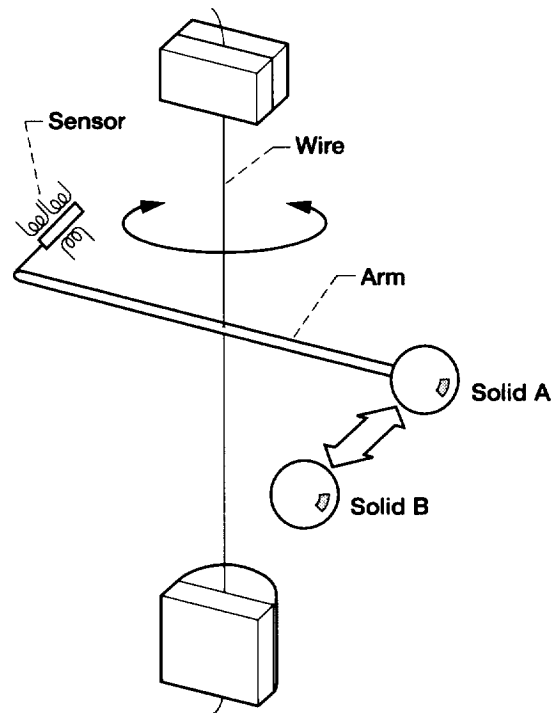


Figure 3.—Schematic diagram of torsion balance adapted from Cavendish balance.

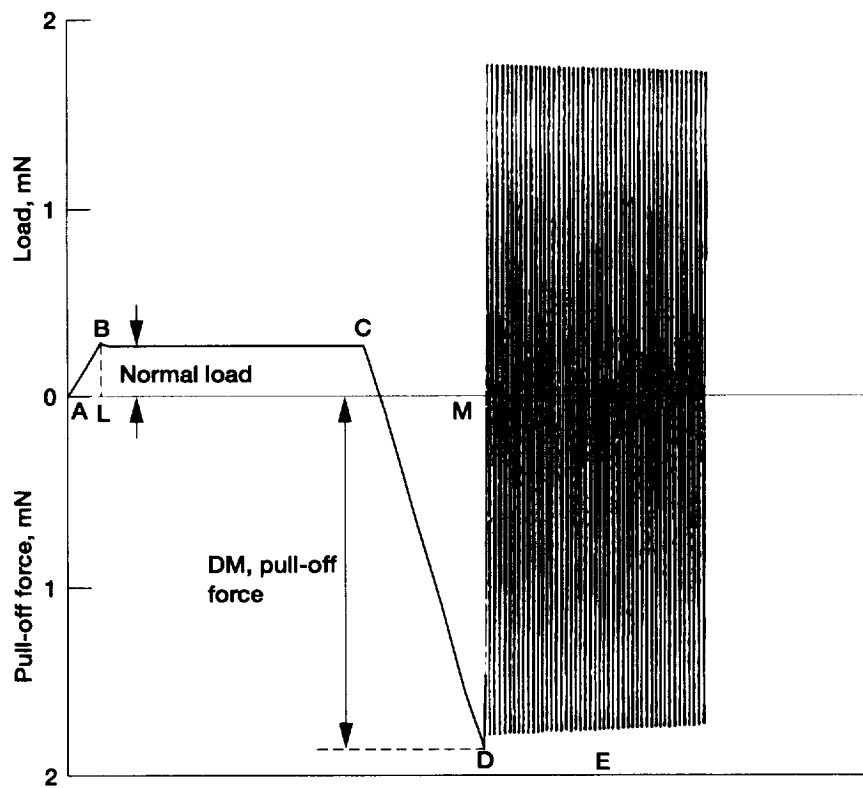


Figure 4.—Typical force-time trace.

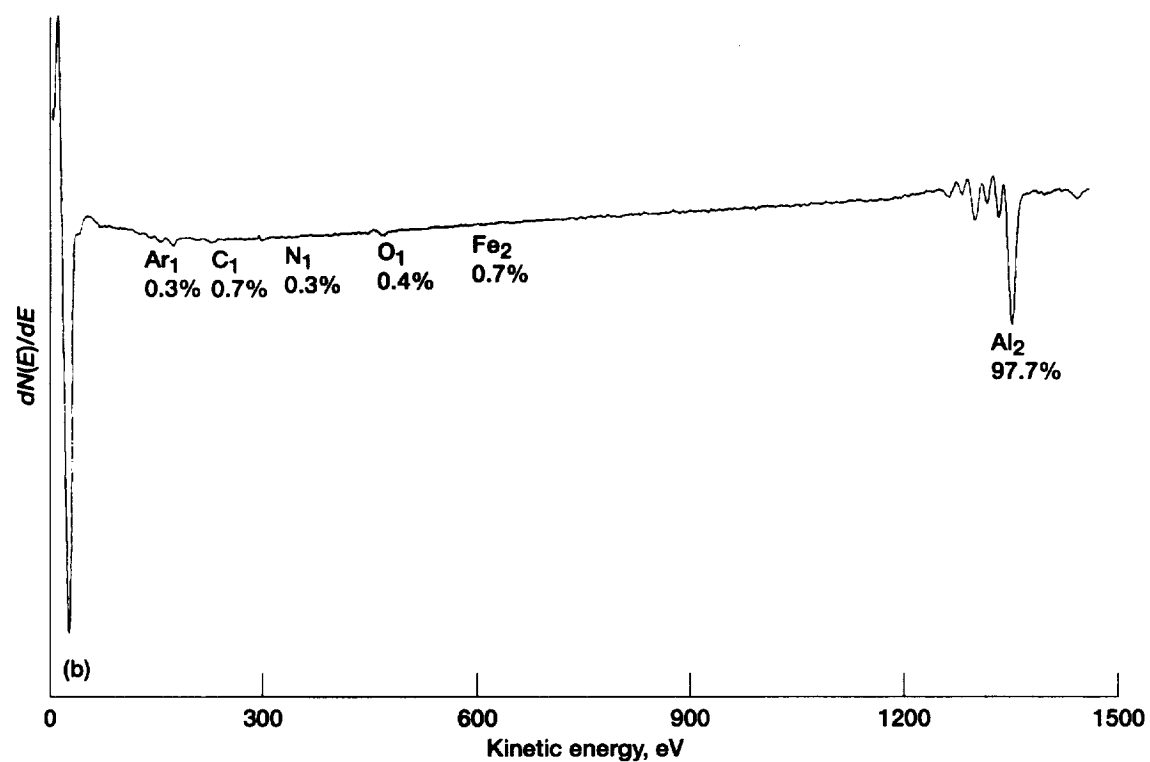
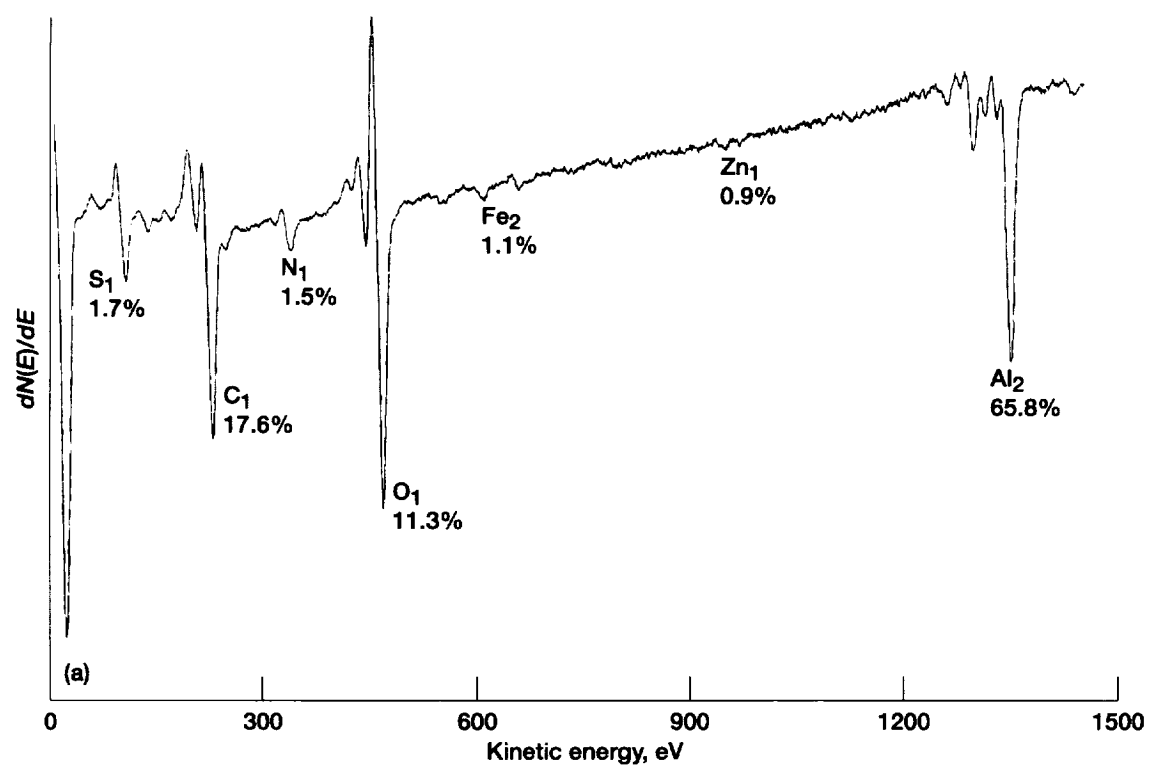


Figure 5.—AES spectra of single-crystal aluminum pin surfaces. (a) Chemically polished surface; 3-keV electron beam. (b) Argon-ion-sputter-cleaned surface; 3-keV electron beam.

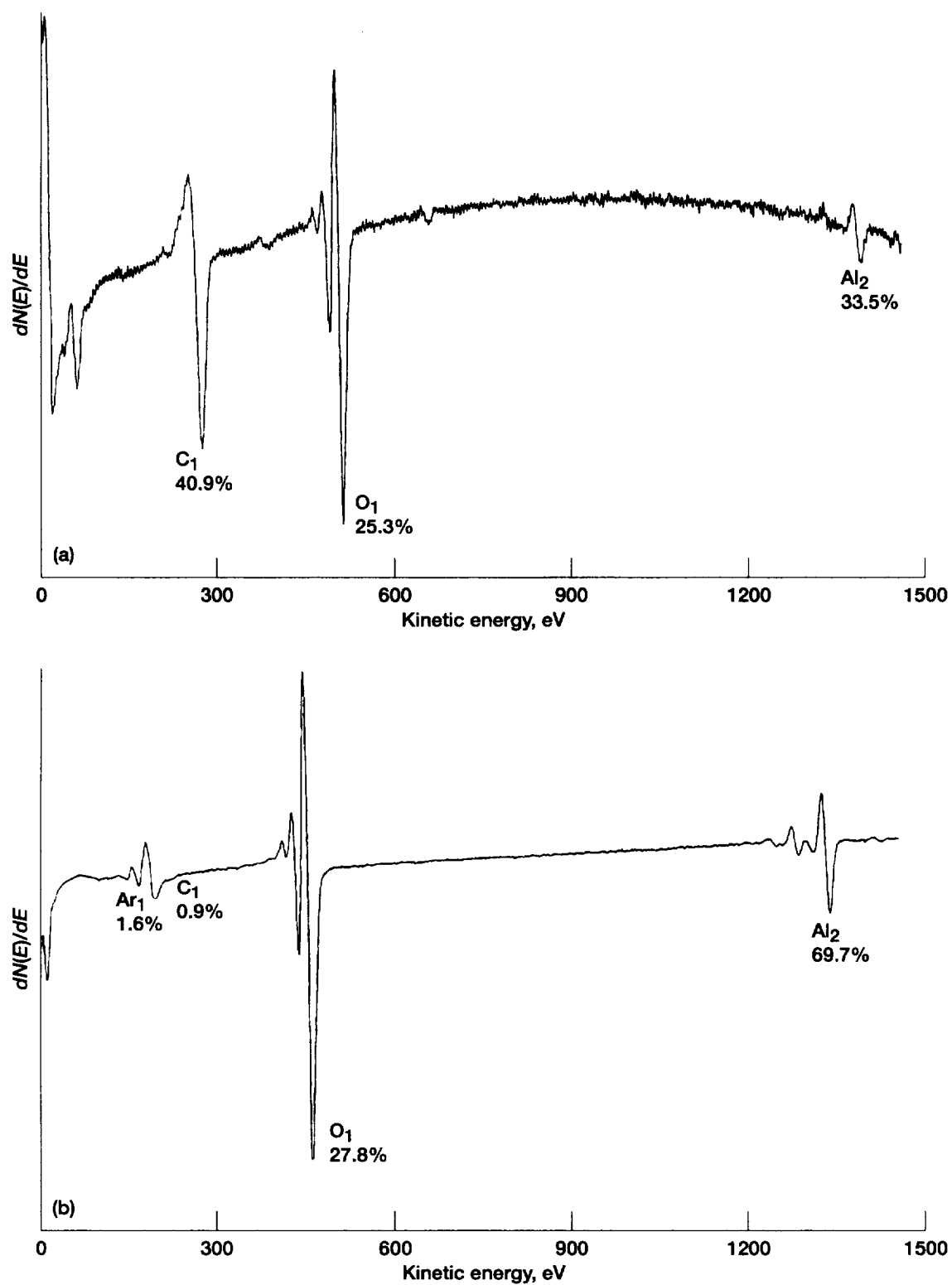


Figure 6.—AES spectra of single-crystal sapphire flat surfaces. (a) As-received surface; 2-keV electron beam. (b) Argon-ion-sputter-cleaned surface; 3-keV electron beam.

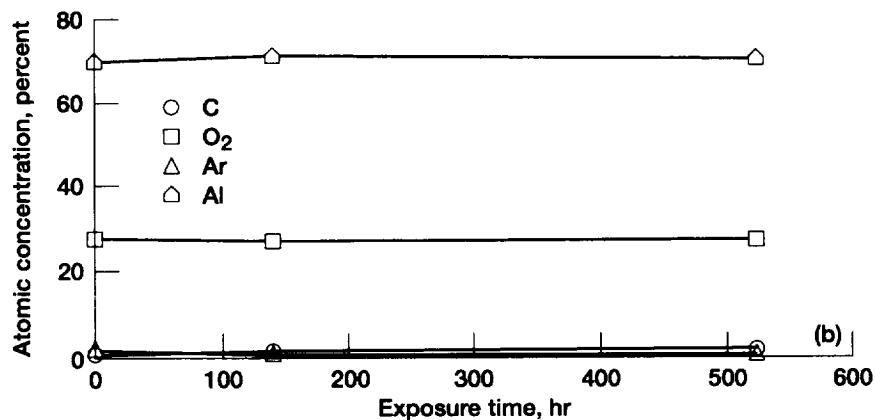
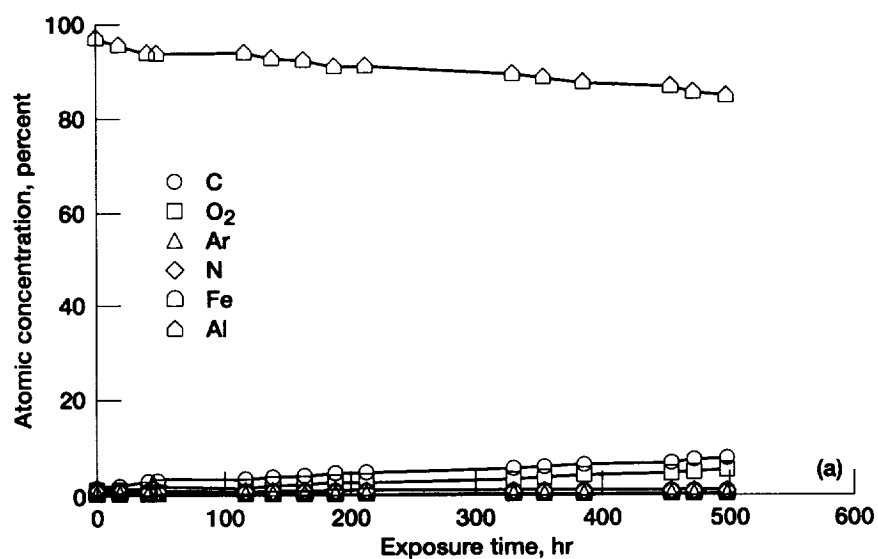


Figure 7.—Element composition as a function of exposure time in ultrahigh vacuum after argon ion sputter cleaning. (a) Single-crystal aluminum pin surface. (b) Single-crystal sapphire flat surface.

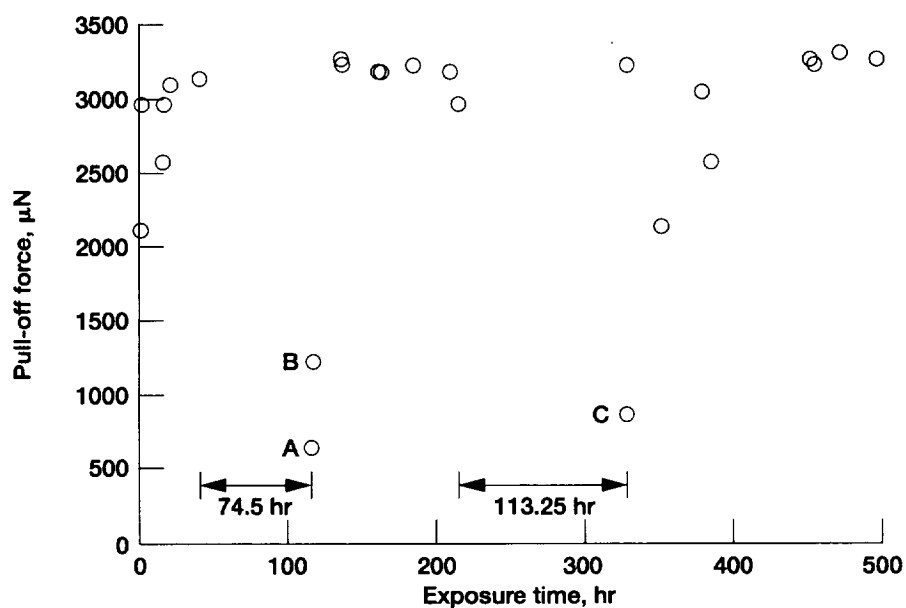


Figure 8.—Pull-off force (adhesion) for clean aluminum pin in contact with clean sapphire flat in ultrahigh vacuum as a function of exposure time after argon ion sputter cleaning. Normal load, 135 μN .

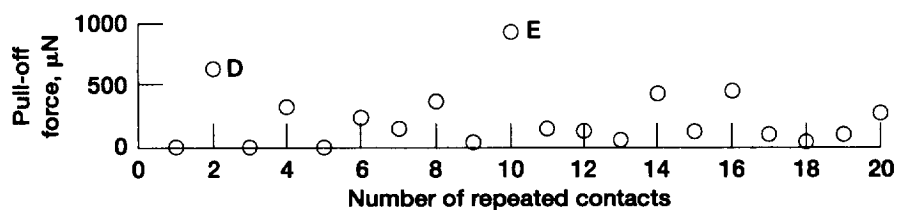


Figure 9.—Pull-off force (adhesion) for contaminated aluminum pin in contact with clean sapphire flat in ultrahigh vacuum as a function of repeated contacts. Normal load, 1063 μN .

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